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#### COMPLETED PROJECT SUMMARY

TITLE: Investigation of Coupled Surface and Bulk Reaction

Phenomena Using CEAPS

PRINCIPAL INVESTIGATOR: Dr. Bruce J. Tatarchuk

Chemical Engineering Department

Auburn University Auburn AL 36849

INCLUSIVE DATES: 1 September 1984 - 30 April 1988

GRANT NUMBER: AFOSR-84-0301

COST AND FY SOURCE: Cumulative Project Funding: \$96,350

(9/1/84 - 10/31/85), \$94,571 (11/1/85 - 10/31/86), \$93,265 (11/1/86 - 10/31/87),

\$48,800 (11/1/87 - 4/30/88)

JUNIOR RESEARCH PERSONNEL: Mr. J.S. Zabinski

Mr. T.S. Lee Mr. J.H. Sanders

### **PUBLICATIONS:**

"Backscatter Mössbauer Spectroscopy: Applications to Surface and Catalytic Phenomena," with J. A. Dumesic, in Chemistry and Physics of Solid Surfaces, Volume 5, Chapter 4, 65-109 (1984). Published by Springer Verlag.

"Electron Intensities Obtained During Backscatter-Mössbauer Spectroscopy: I. Comparison Between Theory and Experiment, "Nuclear Instruments and Methods in Physics Research, B18, 182, (1987), with T.S. Lee, T.D. Placek and J.A. Dumesic.

"Generation of Low Energy Resonant Electrons During Relaxation of <sup>57</sup>Fe," Hyperfine Interactions 41, 737, 1988, with J.S. Zabinski.

"A Theoretical Model for the Analysis of Backscattered-Conversion Electron Mössbauer Spectroscopy: Angular and Energy Distributions," Hyperfine Interactions 42, 1149, 1988, with T-S. Lee.

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Approved for mublic release; distribution unlimited.

"Generation of Low Energy Resonant Electrons During Relaxation of <sup>57</sup>Fe," International Conference on the Applications of the Mössbauer Effect, August, 1987, Melbourne, Australia, Volume III, Proceedings of the International Conference on the Applications of the Mössbauer Effect, J.C. Baltzer AG, Scientific Publishing Co., Basel-Switzerland, 1988, with J. Zabinski.

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"Chemical Characterization of the Deactivation and Protection of FeTi Thin-Films Using Complementary Nondestructive Techniques, "in press, Journal of Thin Solid Films, with J.H. Sanders.

"Resonant Low Energy Electrons and Their Impact on Nondestructive Depth-Profiling of Thin-Film Samples," accepted for publication to Surface and Coatings Technology, with J.S. Zabinski.

"Deactivation Mechanisms for Thin-Film Iron-Titanium Hydrides," submitted to Journal of the Less-Common Metals, with J. Sanders.

"Passivation Mechanisms for Thin-Film Iron-Titanium Hydrides," submitted to the Journal of Physics F: Metals, with J. Sanders.

"Characterization of Reactions Occurring at Buried Interfaces Between Iron and Titanium Thin-Films," in preparation for submission to the Journal of Materials Research, with J. Sanders.

"Generation Mechanisms for Low Energy Electrons Produced During Relaxation of  $^{57}$ Fe and Their Utilization for Depth-Deconvolution," in preparation for submission to Hyperfine Interactions, with J.S. Zabinski.

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"Nucleation and Intercalation of Iron Overlayers on MoS2

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Single Crystal Substrates, " in preparation for submission to Surface Science, with J.S. Zabinski.

"Empirical Procedures for the Depth-Deconvolution of Low Energy Electron Mössbauer Spectra," in preparation for submission to Nuclear Instruments and Methods in Physics Research, with J.S. Zabinski.

#### PRESENTATIONS:

"Applications of Surface Specific Mössbauer Spectroscopy", invited paper at the Symposium on Techniques for the Characterization of Electrode Surfaces, 188th National Meeting of the American Chemical Society, Division of Colloids and Surface Chemistry, Philadelphia, 1984, with J.S. Zabinski and T.R. Nolen.

"Applications of Combined-Backscatter-Conversion Electron and Backscatter-Photon Mössbauer Spectroscopy to Thin-Film Studies," Annual Meeting of the Florida Chapter of the American Vacuum Society, Clearwater, FL, February 1987, with J.S. Zabinski, J.H. Sanders and T-S. Lee.

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"Interfacial Chemical Reactions Between MoS<sub>2</sub> Lubricants and Bearing Materials," Symposium S -- New Materials Approaches to Tribology: Theory and Practice, Fall National Meeting of the Materials Research Society, December, 1988, Boston, with J.S. Zabinski.

"Application and Performance of Silicon-Based Atomic Oxygen Protective Coatings," invited presentation to the 34th International SAMPE Symposium: Tomorrows Materials Today, Session on Space Environmental Effects, May, 1989, Reno, Nevada, with J.H. Sanders, R.T. Booher and P.B. Lloyd.

## THESES IN PROGRESS:

"Theoretical and Experimental Studies of Backscattered-Mössbauer Spectroscopy," T-S. Lee, Ph.D. Dissertation.

"Studies of Ion Beam Induced Mixing and Internal Hydride Formation by Means of Backscattered-Mössbauer Spectroscopy," J. Sanders, Ph.D. Dissertation.

"Significance of Low Energy Resonant Electrons and Photons During Backscattered-Mössbauer Spectroscopy," J. Zabinski, Ph.D. Dissertation.

## GRADUATE STUDENT AWARDS:

Jeffrey Sanders: NASA-Graduate Student Researchers Program Fellowship, 6/16/85 to 6/15/88.

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Jeffrey Zabinski: Southeastern Regional Ph.D. Fellowship in Chemical Engineering, September 1985 to September 1989.

American Vacuum Society - Tennessee Valley Chapter Graduate Student Research Award, 1988-1989.

Travel Grant awarded by the <u>International</u> <u>Congress on the Applications of the Mossbauer Effect (ICAME), Melbourne, Australia, August 1987.</u>

Teh-Shing Lee: Travel Grant awarded by the <u>International</u>
<u>C</u>ongress on the <u>Applications of the <u>Mossbauer</u>
<u>Effect (ICAME)</u>, Melbourne, Australia, August
1987.</u>

## ABSTRACT OF OBJECTIVES AND ACCOMPLISHMENTS:

The influence and manipulation of chemical phenomena occurring at internal interfaces has major technological importance to the Air Force, yet is an area of basic science which has received relatively little attention in the past due to the characterization problems posed by such systems. Better

characterization techniques, providing both nondestructive and depth-profiled chemical and structural insights, are needed so that important issues in: surface chemistry, interfacial adhesion, microelectronics, corrosion, metallurgy, protective coatings, thin-film science, etc., can be accurately diagnosed and subsequently manipulated for technological advantage.

As a result of the above noted realization, we have constructed a device to nondestructively depth-profile thin-film samples from the topmost monolayers to ca. 20  $\mu m$ . The device combines backscatter-conversion electron and backscatter-photon Mössbauer spectroscopy (CEAPS) to provide depth-resolved chemical, electronic, magnetic, and morphological information over this range. An Air Force sponsored study involving the theoretical and experimental development of CEAPS as well as the testing and demonstration of this technique on a number of model systems has been completed. During our efforts:

- o CEAPS has been used to study interfacial reactions that occur between solid-lubricants and bearing materials after vacuum annealing and reactive gas (H2,O2) treatments. The reaction products (FeS, Fe-oxides, FeMo2S4, etc.) affect both the tribological and adhesive properties of the lubricant/bearing system.
- c CEAPS has provided better than monolayer sensitivity for appropriate nuclei and permitted nondestructive analysis from the topmost monolayer to as deep as 20  $\mu m$  into the specimen; and has provided surface information from one monolayer in as short a time as one hour permitting Mössbauer spectra to be collected from clean surfaces in laboratory vacuum for the the first time,
- Monte Carlo simulations of electronic relaxation following nuclear decay have been conducted to identify generation mechanisms for low energy electrons containing surface information,
- o New theoretical treatments for backscattered-Mössbauer spectroscopy have been developed and experimentally verified which accurately predict angular electron distributions, energy distributions and measured signal-to-background ratios thereby allowing more detailed nondestructive depth-profiling of layered substrates. Inclusion of low energy electrons in theoretical models permits the analysis of very thin surface layers, and provides enhanced surface sensitivities,
- CEAPS studies have revealed the chemical and morphological sources of deactivation in FeTi-hydride systems,
- o The above noted deactivation pathways have been eliminated by means of a protective palladium coating, the chemical and structural integrity of the buried-interface (i.e.,

coating/FeTi) has been examined using CEAPS to verify
performance criteria,

- o We have discovered low energy resonant electron signals below 15 eV (i.e., signatures), which are produced in the topmost monolayers of the sample, and which permit easy deconvolution of this layer from the remainder of an infinitely thick substrate. This procedure permits CEAPS to be used in a more complementary fashion with other surface science techniques.
- o CEAPS was used to monitor monolayer equivalent oxidation of substrates with 30 nm silica coatings to verify coating effectiveness for corrosion resistance when exposed to simulated LEO environments,
- o Ion beam mixing of a buried Fe/Sn interface was studied from both Fe Mössbauer and Sn Mössbauer perspectives revealing the intermetallic compounds responsible for the adhesive and corrosion resistant properties of this system,

AFOSR Program Manager: Lt. Col. Larry W. Burggraf Capt. Lee E. Myers

## FINAL TECHNICAL REPORT

Investigation of Coupled Surface and Bulk Reaction Phenomena Using Combined-Backscatter-Conversion Electron and Backscatter-Photon Mössbauer Spectroscopy (CEAPS)

# 1. Objectives and Statement of Work

The influence and manipulation of chemical phenomena occurring at internal interfaces has major technological importance to the Air Force, yet is an area of basic science which has received relatively little attention in the past due to the characterization problems posed by such systems. Better characterization techniques, providing both nondestructive and depth-profiled chemical and structural insights, are needed so that important issues in: surface chemistry, interfacial adhesion, microelectronics, corrosion, metallurgy, protective coatings, thin-film science, etc., can be accurately diagnosed and subsequently manipulated for technological advantage.

As a result of the above noted realization, we have constructed a device to nondestructively depth-profile thin-film samples from the topmost monolayers to ca. 20  $\mu$ m. The device combines backscatter-conversion electron and backscatter-photon Mössbauer spectroscopy (CEAPS) to provide depth-resolved chemical, electronic, magnetic, and morphological information over this range. An Air Force sponsored study involving the theoretical and experimental development of CEAPS as well as the testing and demonstration of this technique on a number of model systems has been completed.

# 2. Status of Research and Accomplishments

Research conducted during the contract period has resulted in the following findings/accomplishments:

- o CEAPS has been used to study interfacial reactions that occur between solid-lubricants and bearing materials after vacuum annealing and reactive gas (H2,O2) treatments. The reaction products (FeS, Fe-oxides, FeMo2S4, etc.) affect both the tribological and adhesive properties of the lubricant/bearing system. [see publication 17 and presentations 5 and 7]
- ceaps has provided better than monolayer sensitivity for appropriate nuclei and permitted nondestructive analysis from the topmost monolayer to as deep as 20  $\mu$ m into the specimen; and has provided surface information from one monolayer in as short a time as one hour permitting Mössbauer spectra to be collected from clean surfaces in laboratory vacuum for the the first time. [see publications 1,6,7,16 and presentation 1]
- o Monte Carlo simulations of electronic relaxation following nuclear decay have been conducted to identify generation mechanisms for low energy electrons containing surface information. [see publications 7,14,16,18 and presentation 5]
- o New theoretical treatments for backscattered-Mössbauer spectroscopy have been developed and experimentally verified which accurately predict angular electron distributions, energy distributions and measured signal-to-background ratios thereby allowing more detailed nondestructive depth-profiling of layered substrates. Inclusion of low energy electrons in theoretical models permits the analysis of very thin surface layers, and provides enhanced surface sensitivities. [see publications 1,2,4,5,8,14,18 and presentations 2,3,5]
- o CEAPS studies have revealed the chemical and morphological sources of deactivation in FeTi-hydride systems. [see publications 9,10,12,13,15 and presentations 4,6]
- o The above noted deactivation pathways have been eliminated by means of a protective palladium coating, the chemical and structural integrity of the buried-interface (i.e., coating/FeTi) has been examined using CEAPS to verify performance criteria. [see publications 9,10,12,13,15 and presentations 4,6]
- We have discovered low energy resonant electron signals below 15 eV (i.e., signatures), which are produced in the topmost monolayers of the sample, and which permit easy

deconvolution of this layer from the remainder of an infinitely thick substrate. This procedure permits CEAPS to be used in a more complementary fashion with other surface science techniques. [see publications 3,6,7,11,16,18 and presentation 8]

- o CEAPS was used to monitor monolayer equivalent oxidation of substrates with 30 nm silica coatings to verify coating effectiveness for corrosion resistance when exposed to simulated LEO environments. [see publication 20 and presentation 8]
- o Ion beam mixing of a buried Fe/Sn interface was studied from both Fe Mössbauer and Sn Mössbauer perspectives revealing the intermetallic compounds responsible for the adhesive and corrosion resistant properties of this system. [see publication 19]

## 3. Jist of Written Publications

- 1. "Backscatter Mössbauer Spectroscopy: Applications to Surface and Catalytic Phenomena," B.J. Tatarchuk and J.A. Dumesic, in Chemistry and Physics of Solid Surfaces, Volume 5, Chapter 4, 65-109 (1984). Published by Springer Verlag.
- 2. "Electron Intensities Obtained During Backscatter-Mössbauer Spectroscopy: I. Comparison Between Theory and Experiment, "Nuclear Instruments and Methods in Physics Research, B18, 182, (1987), T.S. Lee, T.D. Placek, J.A. Dumesic and B.J. Tatarchuk.
- 3. "Generation of Low Energy Resonant Electrons During Relaxation of <sup>57</sup>Fe," Hyperfine Interactions 41, 737, 1988, J.S. Zabinski and B.J. Tatarchuk.
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- 6. "Resonant Low Energy Electrons and Their Impact on Sampling Depth During Backscatter-Electron Mössbauer Spectroscopy," Nuclear Instruments and Methods in Physics Research, B31 576, 1988, J.S. Zabinski and B.J. Tatarchuk.
- 7. "Generation of Low Energy Resonant Electrons During Relaxation of 57Fe," International Conference on the Applications of the Mössbauer Effect, August, 1987, Melbourne, Australia, Volume III, Proceedings of the International Conference on the Applications of the Mössbauer Effect, J.C. Baltzer AG, Scientific Publishing Co., Basel-Switzerland, 1988, J.S. Zabinski and B.J. Tatarchuk.
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- 9. "Surface Chemical Characterization of Internal Interfaces Generated Within Thin-Film Hydrides," Fall National Meeting of the Materials Research Society, December, 1987, Boston, MRS Symposium Proceedings, Microstructure and Properties of Catalysts, Volume 111, 369-374, 1988, J.H. Sanders and B.J. Tatarchuk.

- 10. "Chemical Characterization of the Deactivation and Protection of FeTi Thin-Films Using Complementary Nondestructive Techniques, "in press, Journal of Thin Solid Films, J.H. Sanders and B.J. Tatarchuk.
- 11. "Resonant Low Energy Electrons and Their Impact on Nondestructive Depth-Profiling of Thin-Film Samples," in press, Journal of Thin Solid Films, J.S. Zabinski and B.J. Tatarchuk.
- 12. "Deactivation Mechanisms for Thin-Film Iron-Titanium Hydrides," submitted to Journal of the Less-Common Metals, J.H. Sanders and B.J. Tatarchuk.
- 13. "Passivation Mechanisms for Thin-Film Iron-Titanium Hydrides," submitted to the Journal of Physics F: Metals, J.H. Sanders and B.J. Tatarchuk.
- 14. "Electron Intensities Obtained During Backscattered-Mössbauer Spectroscopy: III. Emergent Energy and Angular Distributions Resulting from Shakeoff and Secondary Events" submitted to Nuclear Instruments and Methods in Physics Research, T.S. Lee, J.S. Zabinski and B.J. Tatarchuk.
- 15. "Characterization of Reactions Occurring at Buried Interfaces Between Iron and Titanium Thin-Films," in preparation for submission to the Journal of Materials Research, J.H. Sanders and B.J. Tatarchuk.
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- 17. "Nucleation and Intercalation of Iron Overlayers on MoS<sub>2</sub> Single Crystal Substrates," in preparation for submission to Surface Science, J.S. Zabinski and B.J. Tatarchuk.
- 18. "Empirical Procedures for the Depth-Deconvolution of Low Energy Electron Mössbauer Spectra," in preparation for submission to Nuclear Instruments and Methods in Physics Research, J.S. Zabinski and B.J. Tatarchuk.
- 19. "Phase Determination and Distribution of an Ion Beam Mixed Fe-Sn Internal Interface," in preparation for submission to Thin Solid Films, J.H. Sanders, D.L. Edwards, J.R. Williams and B.J. Tatarchuk.
- 20. "Measurement of Oxidation Kinetics Through Atomic Oxygen Protective Coatings Using Backscattered-Mössbauer Spectroscopy," in preparation for submission to Thin Solid Films, J.H. Sanders, D. Gulino, B. Banks and B.J. Tatarchuk.

# 4. List of Project Personnel

Dr. Bruce J. Tatarchuk (P.I.) Chemical Engineering Department Auburn University Auburn AL 36849

Mr. J.S. Zabinski (Graduate Research Assistant)
Mr. T.S. Lee (Graduate Research Assistant)
Mr. J.H. Sanders (Graduate Research Assistant)

## THESES IN PROGRESS:

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### GRADUATE STUDENT AWARDS:

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Teh-Shing Lee: Travel Grant awarded by the <u>International</u>
Congress on the <u>Applications of the Mossbauer</u>
Effect (ICAME), Melbourne, Australia, August
1987.

## 5. Presentations

- "Applications of Surface Specific Mössbauer Spectroscopy", <u>invited paper</u> at the Symposium on Techniques for the Characterization of Electrode Surfaces, 188th National Meeting of the American Chemical Society, Division of Colloids and Surface Chemistry, Philadelphia, 1984, J.S. Zabinski, T.R. Nolen and B.J. Tatarchuk.
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# 6. New Discoveries, Inventions and Patents

No patent activities have been pursued. Appropriate discoveries and technical accomplishments are listed in Section 2.